

## REMARKS

This response contains no amendments. Claims 1 and 3-6 remain pending in the case.

In the Office Action of July 14, 2004, the rejection of claims 1 and 3-6 under 35 U.S.C. §103(a) over WO 00/33407 was maintained for the reasons set forth in the Office Action of March 15, 2004, and the rejection was made final. Specifically, the Examiner disagreed with Applicants' assertion that the reference fails to render obvious the use of 1,3-propanediol as a fuel cell coolant. The Examiner stated that the reference specifically teaches that protic liquids such as diols may be used in a fuel cell power generation system, and, "therefore in the absence of showing superior or unexpected results the use of the instantly claimed diol in a fuel cell would have been obvious to one of ordinary skill in the art at the time the invention was made."

1,3-Propanediol (hereafter "PDO"), however, does offer superior and unexpected results as a fuel cell engine coolant relative to other diols. Particularly, PDO is much less easily decomposed to carboxylate acids such as propionate or acetate glycolate and formate under fuel cell coolant system temperature and pressure conditions than ethylene glycol (hereafter "EG"), the diol most likely to be utilized as a fuel cell engine coolant. Exhibit A, attached, shows that a 50% solution of PDO in water produces significantly less formate, propionate and acetate glycolate, and total carboxylate acids than a 50% solution of EG in water when the solutions are maintained under temperature and pressure conditions similar to conditions in a coolant system over a period of time.

Fuel cell engine coolant systems are harmed in two ways by decomposition of a portion of a diol coolant such as EG to carboxylate acids. First, the acidic decomposition byproducts cause the coolant to corrode metal components of the coolant system, leading to premature failure of the coolant system (See, e.g., Exhibit B, A Chemical Base for Engine Coolant/Antifreeze with Improved Thermal Stability Properties, Eator, Boone & Smith, *Soc. of Automotive Engineers, Inc.*, pp 55-61, p. 56 (2001); Exhibit C, Design of Flow Stand to Measure Fuel Cell Coolant Conductivity, Anonymous, *Priorartdatabase.com*, document IPCOM/00028943D (<http://www.priorartdatabase.com/IPCOM/000028943D>), p.1). Aluminum is a common metal component of fuel cell cooling systems that is particularly susceptible to

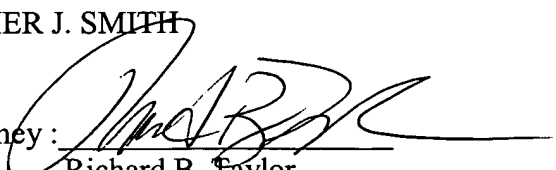
degradation by these acidic decomposition byproducts of EG. In internal combustion engine cooling systems the corrosive nature of EG acid degradation byproducts can be offset by including corrosion inhibitors and pH adjustors. Corrosion inhibitors and pH adjustors, however, cannot be used in a coolant in fuel cell engine cooling systems because they are ionic in nature, and the coolant must have very low conductivity. (See Exhibit C, page 1.)

Second, the acidic decomposition byproducts increase the conductivity of the coolant. The coolant should be as non-conductive as possible to avoid current leakage and short circuiting in the fuel cell. (See Exhibit C, page 1). The acidic decomposition products increase conductivity of the coolant by 1) corrosion and/or leaching of the cooling system materials thus increasing ionic content of the coolant; and 2) the ionic nature of the acidic decomposition compounds themselves.

PDO, therefore, offers superior and unexpected results as a fuel cell engine coolant relative to the most commonly used diol coolant, EG, since PDO is significantly more resistant to degradation into acidic decomposition byproducts under fuel cell engine cooling system operating conditions. Over the course of normal use in a fuel cell engine cooling system, PDO is less corrosive than EG and is less conductive than EG. Exhibit C shows that the acidic degradation byproducts of EG are so serious in a fuel cell engine coolant system that an ion exchange resin filter has been proposed as a solution in such a system to remove the acidic degradation byproducts. The claimed coolant, PDO, is clearly superior to EG in respect to having fewer problems relating to acidic degradation byproducts.

Respectfully submitted,  
EDWARD R. EATON,  
WYNDHAM H. BOON, and  
CHRISTOPHER J. SMITH

By their attorney :



Richard B. Taylor  
Reg. No. 37,248  
Shell Oil Company  
(713) 241-3558

## **EXHIBIT A**

DECLARATION UNDER 37 CFR §1.132



I, Edward R. Eaton, do hereby declare:

1. I am employed by Amalgamated Laboratories, Inc., 2965 W. Osborn Road, Phoenix, Arizona, as President and Chief Engineer.
2. Under my direction and control, Amalgamated Laboratories, Inc. performed testing comparing 1,3-propanediol oxidation stability at high temperatures to that of ethylene glycol under the same conditions without the inclusion of corrosion inhibitors.
3. The test method involved testing 1,3-propanediol and ethylene glycol, respectively, for oxidative stability in an apparatus designed as set forth in American Society for Testing of Materials (ASTM) Standard Test Method D 4340. Oxidative stability was tested by heating a each compound (1,3-propanediol and ethylene glycol) diluted to 50% concentration with non-corrosive water to 300°F for 60 days at a pressure of 28 psi (193 kPa) in the presence of an aluminum specimen, sampling the compounds periodically, and analyzing the samples for formate, propionate and acetate glycolate, and total carboxylic acids—all of which were measured using standard method ASTM D5827—and for pH. In the test, higher concentrations of formate, propionate and acetate glycolate, total carboxylic acids, and lower pH over time indicate less oxidative stability of the tested compound.
4. The test results for oxidative stability comparison between 1,3-propanediol and ethylene glycol from the test method set out in paragraph 3 are attached hereto as Exhibit A1.
5. The test results show that over the course of the test the ethylene glycol solution developed a substantially lower pH, substantially more propionate and acetate glycolate, substantially more formate, and substantially more total carboxylate acids than 1,3-propanediol.

A handwritten signature in black ink, appearing to read "ER Eaton".

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Edward R. Eaton  
Date: 6 December 2004

## **EXHIBIT A1**

*Oxidation comparison between PDO and EG without inhibitors. Test run on D-4340 at 300F, without corrosive water and at 50% concentration.*

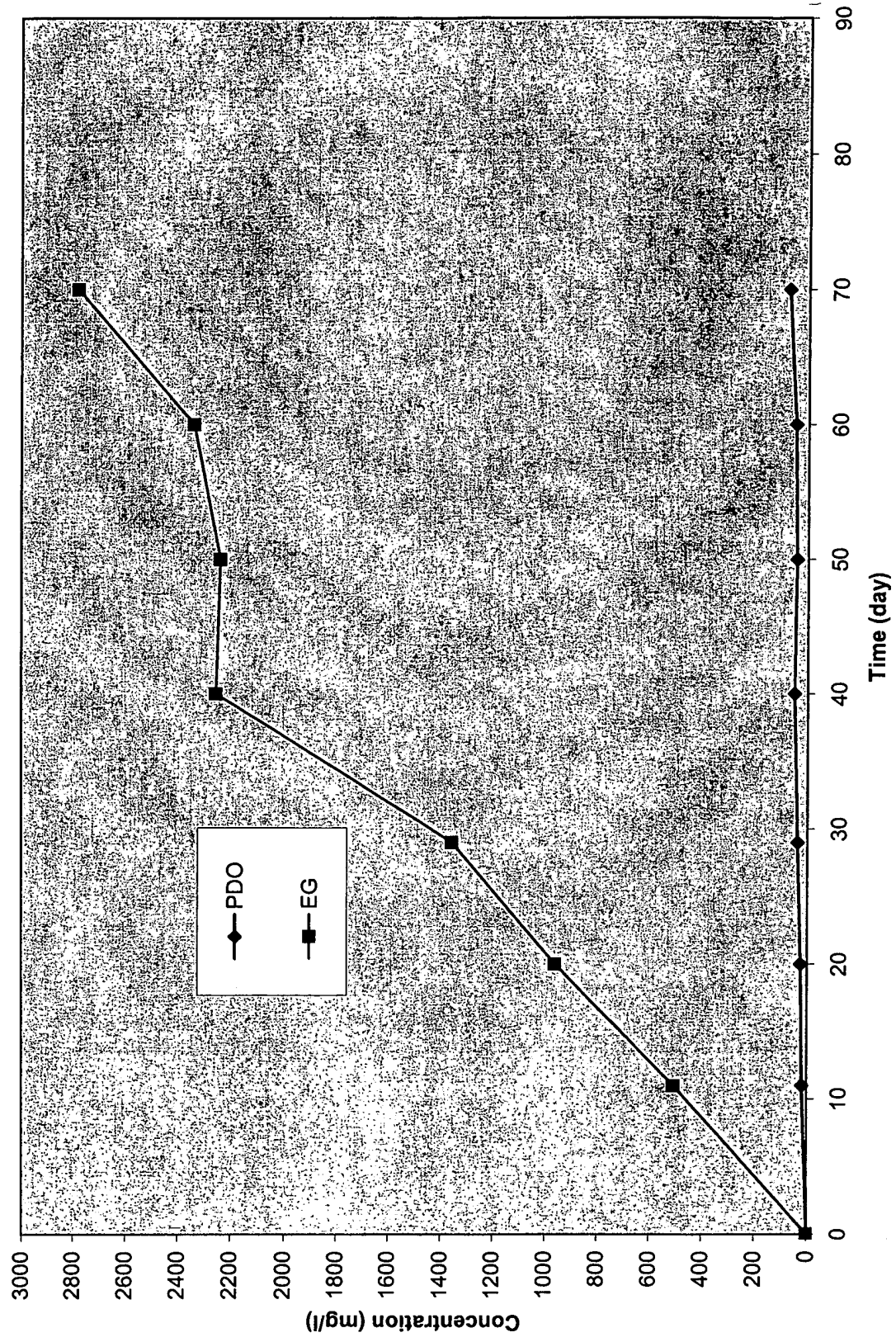
pH										
Time (days)	0	11	20	29	40	50	60	70	81	90
PDO	6.50	4.85	4.74	4.69	4.52	4.61	4.53	4.40		
EG	6.58	3.73	3.49	3.28	3.17	3.10	3.08	3.16		

Propionate&Acetate/Glycolate										
Time (days)	0	11	20	29	40	50	60	70	81	90
PDO	0	19	25	37	50	43	48	73		
EG	0	506	961	1356	2258	2242	2342	2780		

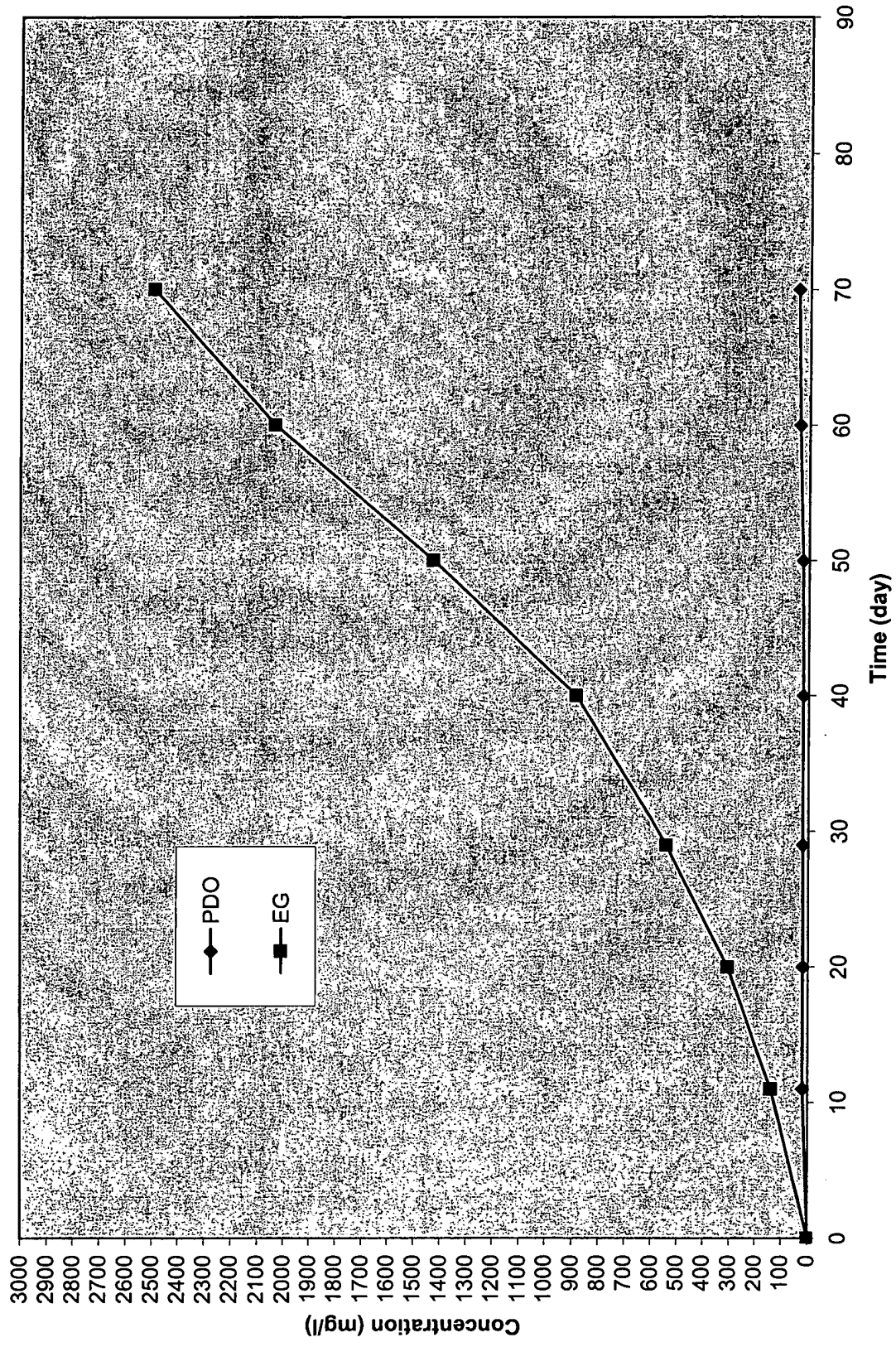
Formate										
Time (days)	0	11	20	29	40	50	60	70	81	90
PDO	0	20	20	22	22	25	38	45		
EG	0	139	307	542	886	1430	2037	2495		

Total Carboxylate Acids										
Time (days)	0	11	20	29	40	50	60	70	81	90
PDO	0	39	45	59	72	68	86	118	0	0
EG	0	645	1268	1898	3144	3672	4379	5275	0	0

# Propionate&Acetate/Glycolate

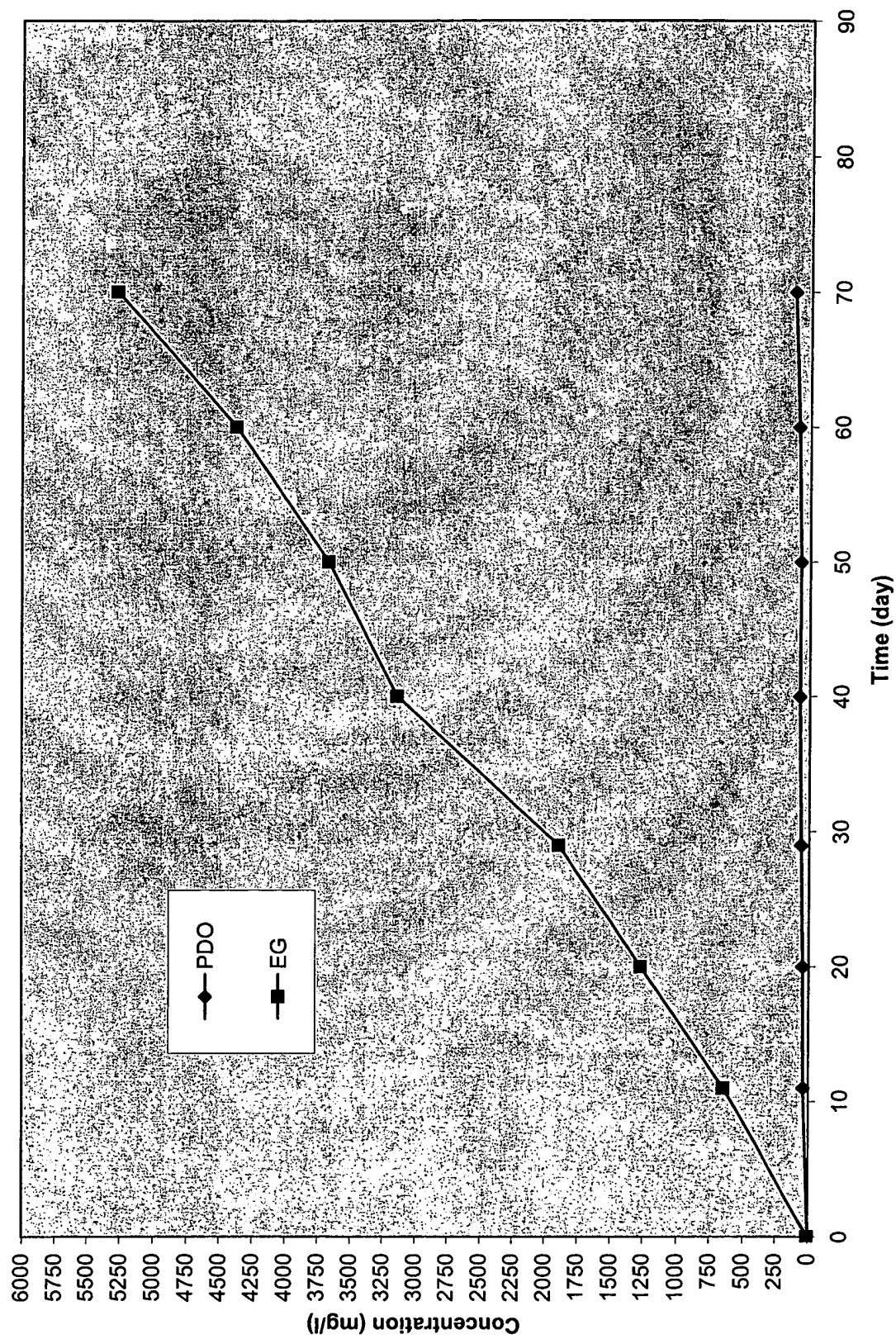


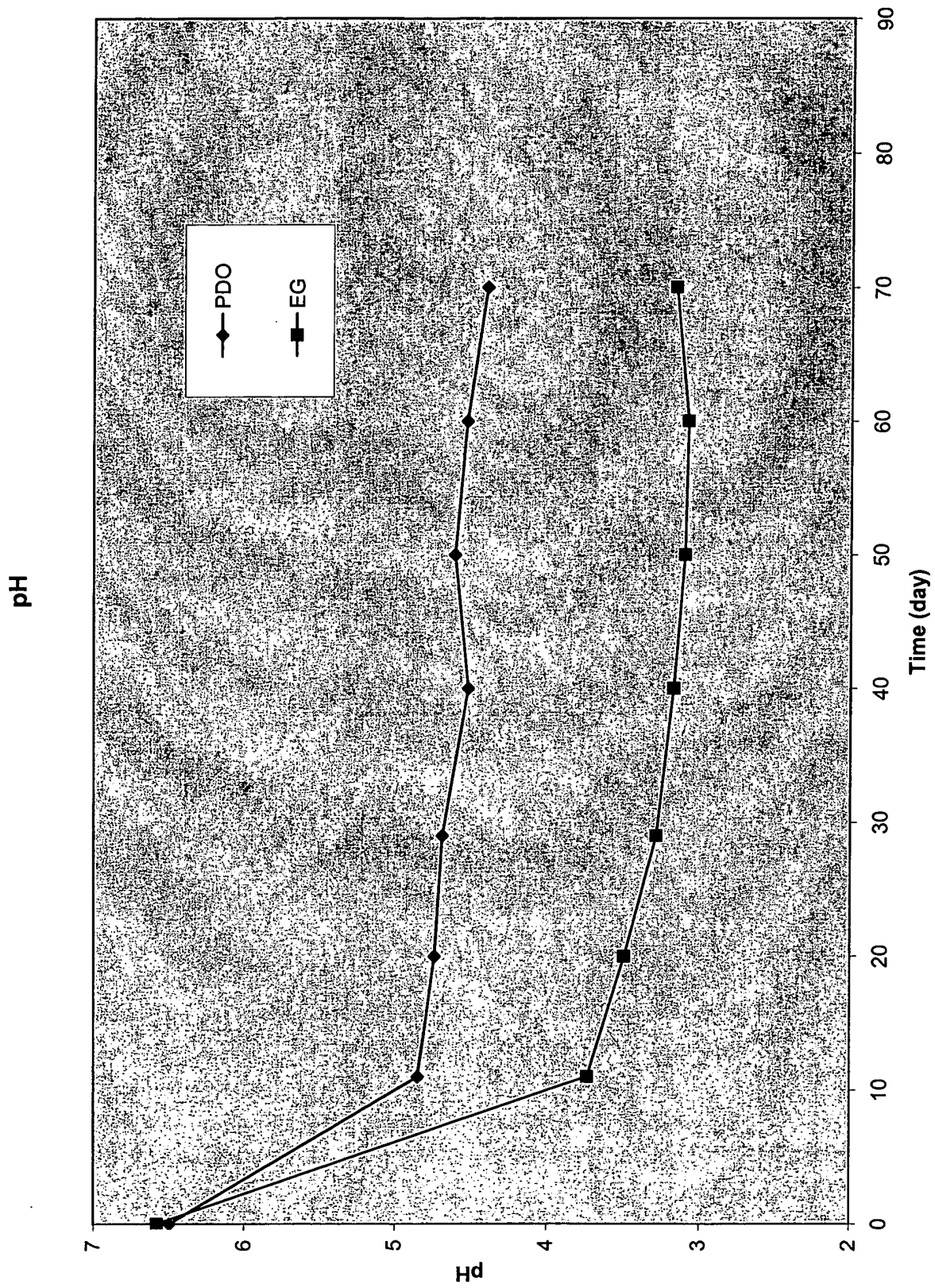
# Formate





# Total Carboxalate Acids





## **EXHIBIT B**

TH18714

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XP-008003886

2001-01-1182

## A Chemical Base for Engine Coolant / Antifreeze with Improved Thermal Stability Properties

P.D. 2001	7
p. 55-61	

Edward R. Eator

Amalgatech Division of Amalgamated Laboratories, Inc

W. H. Boon and Chris J. Smith  
Shell Chemical Co

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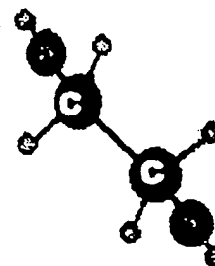
### ABSTRACT

Increasingly challenging international engine emissions reductions have resulted in some advances in engine emissions technologies that may motivate a change from the customary ethylene glycol and/or propylene glycol bases that have been the mainstay of engine antifreeze formulations for almost a century. The new engines' components, especially exhaust gas recirculation (EGR) devices, generate much greater thermal stress on the engine coolant. The oxidation of ethylene glycol and propylene glycol may be accelerated dramatically, resulting in coolant unsuitable for continued use in as little as a few months. The industry has been working towards extended engine coolant service intervals<sup>1,2,3,4</sup>, with some recommendations for service extended to as long as five years. It follows, therefore, that a requirement for coolant change at four to six month intervals (due to accelerated oxidation & aging) would be unacceptable to vehicle owners.

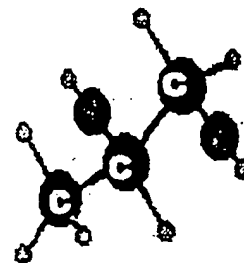
Coolants are generally evaluated and judged by subjecting them to a series of physical properties and performance tests, and then comparing the data to the specifications published by ASTM<sup>5</sup>. This paper reports the data generated by subjecting a new coolant base chemistry, 1,3 Propanediol (PDO), to the ASTM D3306<sup>6</sup> Light and Medium Duty (Automotive) and D6210/6211<sup>7</sup> "Fully Formulated Engine Coolant" physical and performance testing protocols. These protocols qualify an engine coolant for use in virtually any engine cooling system, gasoline (petrol), diesel, and natural gas; engineered with or without wet sleeve cylinder liners.

### INTRODUCTION

Diesel engine OEMs are vigorously working to prepare low-emissions diesel-fueled engines that will meet the emissions requirements of North American and European regulatory agencies in the first decade of the twenty-first century. Extensive investments and research into various possible design improvements have generated some effective technologies. Some of these technologies include the introduction of exhaust gas recirculation devices (EGRs). EGR devices, as the name implies, pass part of the exhaust gas back to the combustion chamber to lower target emissions. Exhaust gases, especially those produced by a diesel engine, are much hotter than regular intake air. The EGR gases, therefore, must be cooled by the existing engine coolant system before they reach the



Ethylene Glycol  
(1,2 Ethanediol)



Propylene Glycol  
(1,2 Propanediol)

Figure 1.1: EG & PG

**Figure 2: Freeze Point Characteristics of PDO Compared to EG-Based Coolant (°C).**

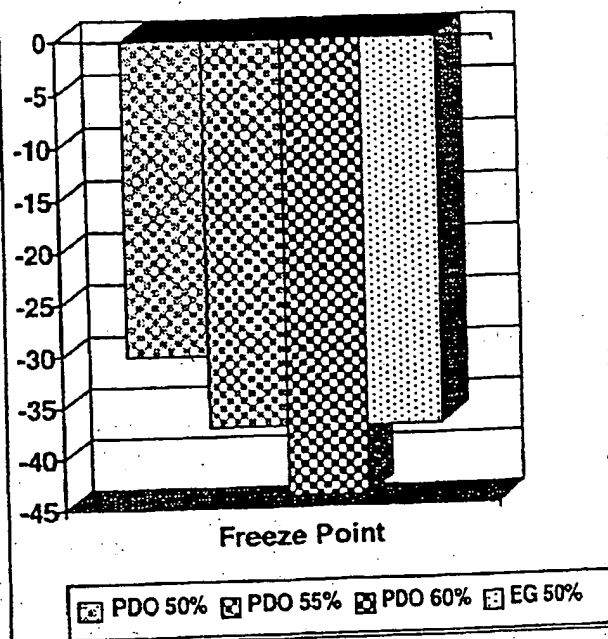


Figure 2 illustrates the aqueous solution freeze point characteristics of the GM 6043 PDO and GM 6043 EG.

**Table 1.1 Physical Tests** There is a slight compromise of the freeze protection as determined by the ASTM D1177<sup>9</sup> test method, but the scientist performing the test noted that the PDO was distinctly softer and more wet in the characteristically slushy phase of diols at the determined freeze point. This could be an indication that actual protection against hard, damaging freezing is actually better, approaching the effective protection point of the EG-based product. The authors also performed the D1177 test with 55% and 60% PDO in water, and found that the 55% concentrated product offered protection equivalent to 50% EG, per the test method. Freeze protection continued to improve at 60% PDO. The authors feel that the antifreeze properties of the chemistry are acceptable; indeed a 50% solution would provide adequate protection against freezing in most geographies. Table 1.1 shows the following:

The boiling point of the 50% solution by ASTM D1120 is 106°C, one degree lower than the 50% EG at 107°C. PDO has a slightly lower specific gravity (SG) than EG when measured by the ASTM D1122 method. The EG based antifreeze was found to have a SG of 1.129 whereas the PDO SG was measured at 1.070. Neither coolant had any effect when subjected to the effect on automotive finish evaluation (ASTM D1882). Indeed, as

**Table 1.1: Physical Properties: Light Duty**

Test Number & Description	PDO 6043	GM 6043 (EG)
D1122 Specific Gravity	1.070	1.129
D1177 Freeze Point 50% vol. in water	-28 °C (-18 °F)	-38 °C (-36 °F)
D1120 Boiling Point 50% vol. in water	106 °C (222 °F)	107 °C (226 °F)
D1882 Auto Finish Effect	none	none
D1119 Ash Content	0.81 %	0.87%
D1287 pH: 50% vol. in DI water	9.8	8.67
D3634 Chloride	16	11
D1123 Water, mass percent	4.1%	4.0%
D1121 Reserve Alkalinity	9.8	9.3
D1881 Foaming Tendencies	Break 3.6 Sec Vol. 113 ml	Break 1.6 Sec Vol. 50 ml

**Table 1.2: Glassware Testing Data Light Duty**

Test Number & Description	PDO 6043	GM 6043 (EG)
D4340 Corrosion of Aluminum Heat Rejecting Surface	0.27 mg/cm <sup>2</sup> /week	0.12 mg/cm <sup>2</sup> /week
D1384 Corrosion in Glassware	Cu 3 mg Solder 1 mg Brass 2 mg Steel 1 mg Cast Fe 2 mg Cast Al 0 mg	3 mg 1 mg 2 mg 1 mg 1 mg 0 mg

**Table 1.3 Simulated Service Tests Light Duty**

Test Number & Description	PDO 6043	GM 6043 (EG)
D2570 Simulated Service	Cu 3 mg Solder 0 mg Brass 7 mg Steel 1 mg Cast Fe 0 mg Cast Al 4 mg	5 mg 4 mg 0 mg 3 mg 3 mg 1 mg
D2809 Water Pump Cavitation-Erosion	8	8

reflected in tables 1.1 – 1.3, 2.1 to 2.3 and 3.1 to 3.3, many of the physical properties of the two coolants are very similar. The similarity is beneficial, because the new coolant will almost certainly be contaminated from time to time with EG and/or PG based coolants. When contamination occurs, it would be desirable that no harm

The data are interesting in that, in this set of tests, two annoying tendencies of "type A" formulations are soundly dampened by the PDO. Namely, higher solder corrosion in D1384 and D2570 and the inconsistent water pump erosion performance sometimes seen in the D2809, as was reported in this sequence.

An additional test was engineered. As coolant ages in use, some of the glycols oxidize. Samples of the control 'type A' and PDO 'type A' coolants were blended at 50% and heated at 150 °C for 28 days in an ASTM D4340 test rig (no corrosive water was added). Ethylene glycol may form formic acid, glycolic acid or oxalic acid. PDO was analyzed for these and larger C<sub>3</sub> carboxylates, in case degradation of the PDO molecule might produce them.

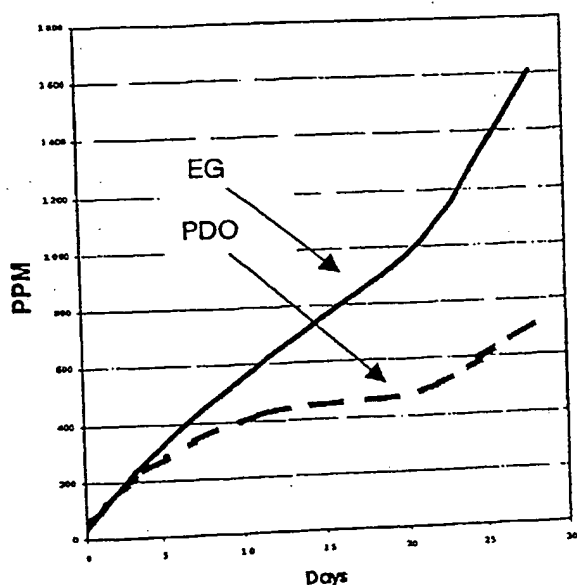


Figure 3: Oxidation Products vs. Time

Figure 3 illustrates the behavior of the coolants' degradation acids. This data suggest that high temperature characteristics of PDO may be better, that it may resist oxidation degradation better than EG.

Figure 4 illustrates the behavior of nitrite. It is intriguing to observe that the nitrite concentration is extremely stable in the PDO while the nitrite manifests a classical and expected oxidation to nitrate in EG. There are no metals in this test; pure oxidation is the only depletion mechanism.

With type 'B' inhibitor, PDO offers equivalent to slightly better corrosion protection evidenced by modified D1384 and D2570 data. In particular, chemical analyses of the coolants were conducted to quantify and compare the degradation of coolants as evidenced by the concentration of oxidation products.

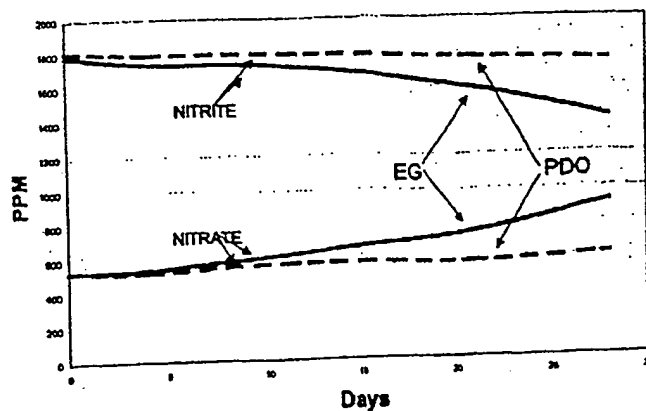


Figure 4: Nitrite and Nitrate Behavior

PDO type 'A' and type 'B' both produced very positive data. In the higher temperature testing, data either were equivalent to or better than the EG. Significant improvements were documented in lead solder performance, aluminum water pump erosion/corrosion and degradation of PDO. Corrosion in glassware, the ASTM D1384 method, was performed with the standard set of six metal samples. Type 'B' data for each the six metals are reported in table 2.2. The standard test requires 336 hours of exposure at 88 °C. This test was run for 336 hours at 150 °C by using a propylene glycol bath instead of water. Similarly, The coolant temperature was elevated in the D4340 apparatus.

The maximum safe operating temperature of the standard simulated service rig was deemed to be 93°C, five degrees warmer than the normal operating temperature of 88°C. In general, the data from the simulated service paralleled that of the corrosion in glassware test.

Finally, the two coolants were evaluated in a hot surface scale rig. There are currently no ASTM requirements or suggestions for limits in scale formation resulting from this method. The proposed method involves introducing a fluid consisting of 8 vol% of the sample mixed in corrosive hard water into the test apparatus. The apparatus is operated for 100 hours, exposing the sample to a hot steel surface in order to learn if the chemistry can prevent the formation of scale. The control 'type A' performed somewhat about the same as the PDO 'type A'. The EG control generated 1.6 grams of scale and the PDO 2.3 grams. This data suggests both coolants would require additives to prevent scale formation in service with hard water, although the 'type A' formulation technology has exhibited positive fleet experience.

## CONCLUSIONS

A prospective new base chemical has been discovered that resists oxidation due to thermal stress better than ethylene glycol. The chemistry, Shell Chemical's 1,3 propanediol or PDO, may be successfully inhibited with conventional light duty and fully formulated heavy-duty inhibition technologies. The coolant base has passed all of the high temperature-modified ASTM type tests, demonstrating PDO's capabilities in severe operating environments.

Continuing work to certify the chemistry to existing ASTM engine coolant performance standards is being conducted, but the coolant is expected to pass, given that it has already succeeded under more severe test conditions. Inhibitor package optimization for use in PDO may offer additional benefits in the final development of an extended service coolant technology.

Another phase of research should investigate the behavior of PDO inhibited with extended service inhibitor chemistry, such as that used in the Caterpillar and Texaco extended service products based on 2-ethylhexanoic acid and sebacic acid carboxylate inhibitors. It will also include various 'hybrid' formulations such as are preferred by John Deere, Ford and Daimler-Chrysler.

## ACKNOWLEDGMENTS

The authors appreciate the contributions of Amalgatech, The Penray Companies, Inc., Dober of Glenwood, Inc. and especially Shell Chemical Company in making this research possible.

## REFERENCES

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3. Sarkis S. Aroyan and Edward R. Eaton, "Fleet Test Evaluation of Fully-Formulated Heavy-Duty Coolant Technology Maintained with a Delayed-Release

Filter Compared with Coolant Inhibited with a nitrated Organic Acid Technology", *Engine Coolant Testing*, Fourth Volume, ASTM STP 1335, R. E. Beal, Ed., American Society for Testing and Materials, West Conshohocken, PA, 1997

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5. *Engine Coolants, Halogenated Organic Solvents and Fire Extinguishing Agents; Industrial and Specialty Chemicals*, Standards on Disk, American Society for Testing and Materials, West Conshohocken, PA, vol 15.05, 2000 D3306-00
6. *Ibid* D4985-00
7. *Ibid* D6210 and D6211
8. GM 6043 Standard Coolant Formulation, General Motors Corporation, Detroit, MI
9. ASTM Standards on Disk, D1177-94

## CONTACT

Edward R. Eaton, Amalgatech Division, Amalgamated Laboratories, Inc. 13901 N 73<sup>rd</sup> St, Scottsdale, AZ 85260  
Telephone: 480-556-0888, Fax: 480-991-2903; e-mail: [ereaton@amalgatech.com](mailto:ereaton@amalgatech.com)

## DEFINITIONS, ACRONYMS, ABBREVIATIONS

ASTM – American Society for testing of Materials

EG – Ethylene Glycol (1,2 ethanediol)

EGR – Exhaust Gas Recirculation device

GM – General Motors Corporation

OEM – Original Equipment Manufacturer (s)

PDO – (1,3 propanediol)

PG – Propylene Glycol (1,2 propanediol)

## **EXHIBIT C**



## **Design Of Flow Stand To Measure Fuel Cell Coolant Conductivity**

Fuel Cell Vehicle Cooling System requires coolant of very Low Electrical Conductivity: The unique coolant should not have corrosion inhibitors, which are ionic, and therefore the coolant electrical conductivity will increase from the leaching of ions of cooling system components. It is unknown about how much ions leach out of components used in a Fuel Cell Vehicle cooling system. This information is necessary to design the Fuel Cell Vehicle Cooling System.

A unique Flow Stand outside of the vehicle had to be designed which could allow balanced flow of coolant through the cooling system components at controlled conditions in order to find the electrical conductivity of the unique coolant flowing through the cooling system components.

### **Description**

Traditional internal combustion engine coolant formulations primarily consist of water and ethylene glycol. Although water and ethylene glycol themselves do not conduct very well, these formulations will not meet the low conductivity requirement because additives, such as corrosion inhibitors, pH adjustors, and dyes, are ionic in nature, thereby increasing the conductivity of the coolant. Therefore, the coolant selected for the Fuel Cell Vehicle is a mixture of deionized water and ethylene glycol with no additives. However, this only ensures initially low coolant conductivity. Ethylene glycol decomposes in the presence of oxygen to form acidic by-products, such as glycolic, glyoxylic, and formic acids. The presence of these by-products not only increases the conductivity, but also can significantly accelerate corrosion processes in the coolant loop due to the lack of corrosion inhibitors and pH buffers. Further, the rate of ethylene glycol decomposition increases with increasing temperature.

In addition, plastic, aluminum, and rubber components used in the cooling system add ions (organic and inorganic) into the coolant through leaching, degradation and/or corrosion processes, increasing conductivity.

The unique coolant in the Fuel Cell Vehicle (FCV) is a mixture of deionized water/ethylene glycol. For functioning of the Fuel cell, it is required that the coolant that flows through it should maintain very low electrical conductivity ( $<5 \mu\text{S/cm}$ ) to avoid current leakage and short circuiting, presenting a unique water chemistry issue. The coolant's initially low conductivity increases as: 1) ions are released from system materials through leaching, degradation and/or corrosion, and 2) organic acids are produced by ethylene glycol degradation. Estimating the electrical conductivity of the coolant as it flows through the components of the cooling system is very important for design and operation of fuel cell vehicles. The unique Flow Stand apparatus was designed and built to replicate the vehicle like conditions of the coolant and study the effect of the coolant flow through various cooling system components.

An on-board ion exchange resin filter, containing both anion and cation exchange resins, has been used to remove unwanted ions by exchanging them with  $\text{H}^+$  or  $\text{OH}^-$  and therefore maintain low coolant electrical conductivity. The unique Flow Stand apparatus (figure 1) was designed to meet the following vehicle requirements:

- a. Coolant should flow at maximum flow rate of 120 liters per minute through the components
- b. Coolant should be able to be heated to at least a maximum vehicle temperature of 80 deg. Celcius
- c. Part of the full flow go through the De-Ionized (DI) Filter
- d. DI Filter should be at the downstream of the pump
- e. Particle Filter should be at the downstream of the test section components

The unique Flow stand apparatus was designed with the above requirements and following design considerations were added to help aid the testing of the coolant, data analysis and easy maintainability.

- a. The Flow Stand should be able to record electrical conductivity at the rate of 1 data per minute
- b. The Flow stand should have the on board DI-Filter and Particle Filter like the vehicle does
- c. The Conductivity Sensors CS-1, CS-2 (figure 2) should be positioned at the inlet and outlet of the test component flow path
- d. The test components should be easily removal between the inlet and the outlet
- e. The amount of flow going through the DI-Filter, ion exchanger (figure 2) should be the at the maximum 5 % of the main stream flow just like the vehicle is designed
- f. The Coolant fill bottle, i.e Fill Tank (figure 2) cap could be easily opened and closed and opened to the atmosphere for worst case conditions to be tested
- g. The Coolant could be easily removed from the system, outlet sample port (figure 2) in closed vials for water chemistry analysis
- h. There was not too much noise generated by the running of flow stand pump
- i. The components of the flow stand for example the plumbing 316 stainless steel (figure 2) that were not vehicle components did not introduce any variation in the test data
- j. The flow stand should be able to drain easily, system drain (figure 2)
- k. The flow stand should be able to bleed air to a tank like the degas bottle does in a vehicle, air bleed to tank (figure 2)
- l. The Flow stand should have a safety feature for Pressure Shut Off
- m. The flow stand should have a safety shut off switch

The schematic of the Flow stand is shown in Figure 2. In order to meet the flow requirements a 3 HP, 3500 RPM, 240VAC 3-phase, inverter duty motor pump (103 MP) was chosen. 316 Stainless Steel head and plumbings were passivated before use to eliminate the leaching of ions from these components. Conductivity sensors (219CS-1,2) were rated for 0-100 microsiemens with a 0.05 cell constant. The Flow meters (411FM) used were turbine type with capacity to measure 0-60 gallons per minute. All the ball valves (MV) were stainless steel. The DI-Filter (FIL02) which is a mixture of anion and cation resin had a capacity of 1 liter. The Particle Filter (FIL01) has a mesh element 50x250 plain Dutch weave and is Stainless Steel to prevent its leaching with the coolant.

The diameter of the plumbing used in the mainstream flow was 1.5 inch and the material was 316 Stainless Steel. 0.75 inch diameter 316 Stainless Steel plumbing was used in the by pass line for coolant going through the ion exchanger. These sizes allowed for coolant not to choke in the DI-Filter.

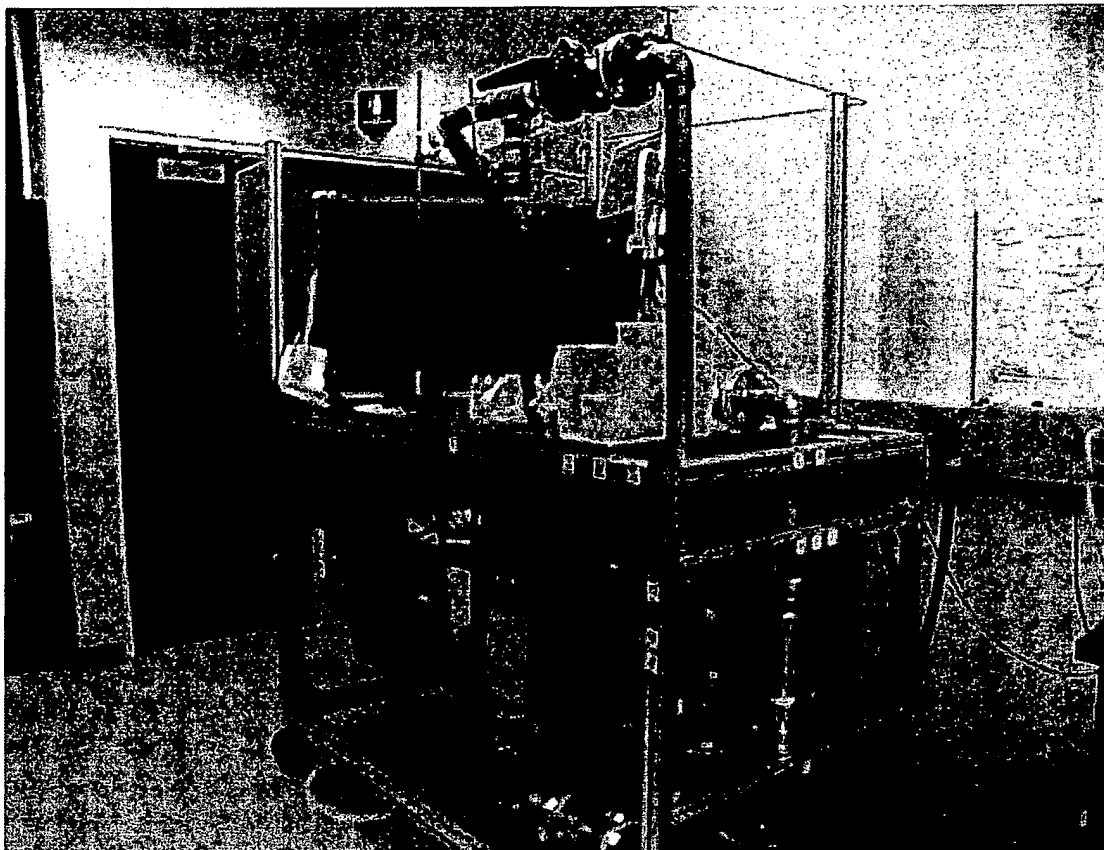


Figure 1

The test data shown in figure 3 shows the coolant electrical conductivity over time for flow of coolant going through the two heat exchangers, namely the High Temperature Radiator and the Heater core. Worst case condition were tested which was a flow of 120 liters per minute and the coolant temperature was 80 degrees celcius. These conditions allow for maximum leaching on ions from test components into the coolant. The Test data for 500 hours of continuous use of the flow stand shows that the coolant conductivity never increased over the specified requirements with the components used in the flow stand. The size of the DI filter was chosen to meet the vehicle requirement.

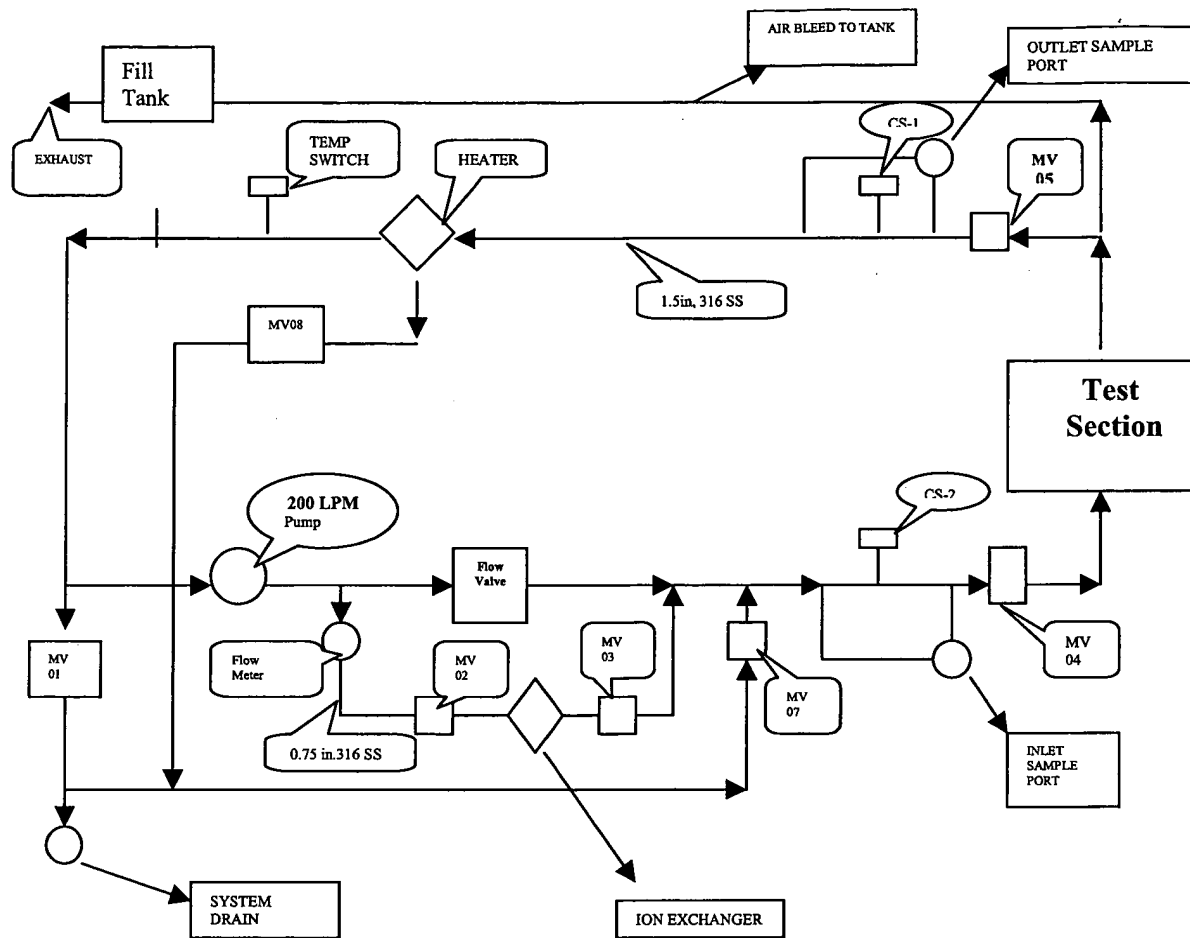


Figure 2

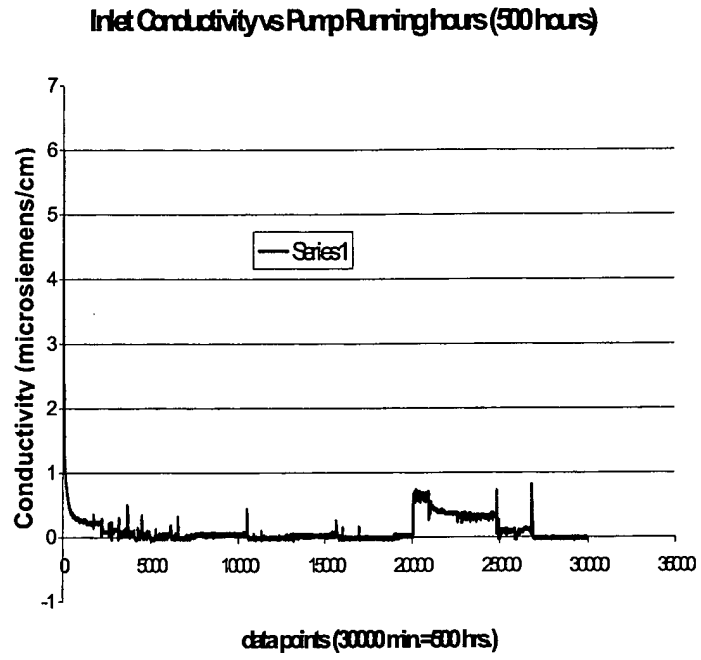


Figure 3

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